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IS 12860 (1989): Metallic Coating Thickness By X-ray Fluorescence Technique Method - Determination [MTD 21: Non-Destructive Testing]

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Indian Standard

METALLIC COATING THICKNESS BY X-RAY FLUORESCENCE TECHNIQUE METHOD— DETERMINATION

भारतीय मानक

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Non-destructive Testing Sectional Committee, MTD 21

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 8 December 1989, after the draft finalized by the Non-destructive Testing Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard has been prepared as a guide for quick determination of metallic coating thickness by X-ray fluorescence technique on the shop floor. A typical application of this method is measurement of zinc coating of galvanized sheets, as hot dipped galvanized sheets of mild steels are in great demand in day-to-day applications. Galvanizing of mild steel sheets is done to prevent corrosion, but excessive coating leads to adverse effect on weldability characteristics. Thus coating mass has to be maintained within specified limits so that they are acceptable to customer requirements. Method of measuring the coating thickness of galvanized sheets has been specified in IS 6745 : 1972 'Method for determination of mass of coating on zinc coated iron and steel articles' . Recognizing the need for a quick shop floor test, this standard has been prepared to provide a guide for procedure to be adopted for rapid determination of coating thickness of galvanized sheets by XRF techniques.

In reporting the results of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METALLIC COATING THICKNESS BY X-RAY FLUORESCENCE TECHNIQUE METHOD — DETERMINATION

1 SCOPE

1.1 This method covers the use of X-ray Fluorescence (XRF) techniques for determining the thickness of metallic coatings. This technique is applicable to stationary as well as moving samples.

1.2 This standard is applicable to the determination of mass of coating per unit area as well as coating thickness. The general principles presented here are applicable to the determination of the thickness of most metallic coatings on any substrate, metallic or non-metallic. The maximum measurable thickness for a given coating is that thickness beyond which the intensity of the characteristic secondary X-rays no longer changes by a significant amount with increase in thickness.

1.3 Personnel Protection

For the purpose of safety of personnel IS 2598 : 1966 shall be referred.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
2598 : 1966	Safety code for industrial radiography practice
6745 : 1972	Method for determination of mass of zinc coating on zinc coated iron and steel articles

3 PRINCIPLE

3.1 The measurement of the thickness of coating by XRF method is based on the combined interaction of the coating and substrate with an intense beam of primary radiation. The primary radiation may come from a radioisotope source or be generated by an X-ray tube. This interaction results in the generation of secondary X-rays of well defined energy. Each element fluoresces at an energy that is characteristic of the element alone. (For best results the difference in atomic number of coating material to that of the base metal should at least be two.)

The intensity of the selected energy is measured by means of an appropriate radiation detector in conjunction with electronic pulse amplitude discriminator and pulse counting (scaler) circuitry.

A relationship exists between coating thickness and intensity of secondary radiation up to the limiting thickness mentioned in **1.2**. Measurements are based on the use of primary standards of known coating thicknesses which serve to correlate quantitatively the radiation intensity, thickness and density.

4 TECHNIQUES

4.1 Coating Thickness Measurement by Emission of Fluorescent X-Rays

In this technique the detection system is set up to count the number of X-rays in the energy region of characteristic X-rays of specific metal of interest (Zinc in case of galvanized sheets and tin in case of tin plates). The count rate intensity at this energy will be minimum for a sample of the bare substrate where it consists only of scattered (back ground) radiation. For a sample having coating thickness beyond the saturation limit conditions the count rate is maximum. For a sample having coating thickness intermediate to the above limits, the intensity will have an intermediate value. The intensity of the secondary X-radiation depends in general upon the excitation energy, the atomic numbers of the coating and substrate, the area of the specimen exposed to the primary radiation and the thickness of coating. If all other variables are fixed, the intensity of the characteristic radiation is a function of the thickness or mass per unit area of the coating. The exact relationship between the measured intensity and the corresponding coating thickness shall be established by the use of standards having similar coating and substrate composition as the sample to be measured. The limiting thickness depends upon the atomic number of coating and the primary radiation energy. The typical relationship between a coating thickness and the intensity of characteristic emission from the coating material is illustrated by the curve in Fig. 1.

4.2 Thickness Measurement by X-Ray Absorption

In this technique the intensity of secondary X-radiation emitted from base material is recorded. The intensity will be maximum for a sample of the uncoated material and will decrease with increasing coating thickness. This is because the existing and secondary radiation undergo attenuation

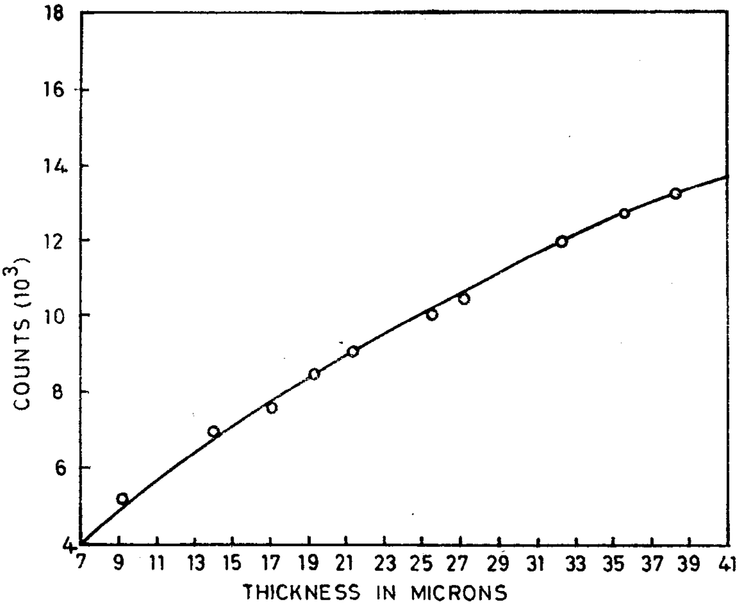
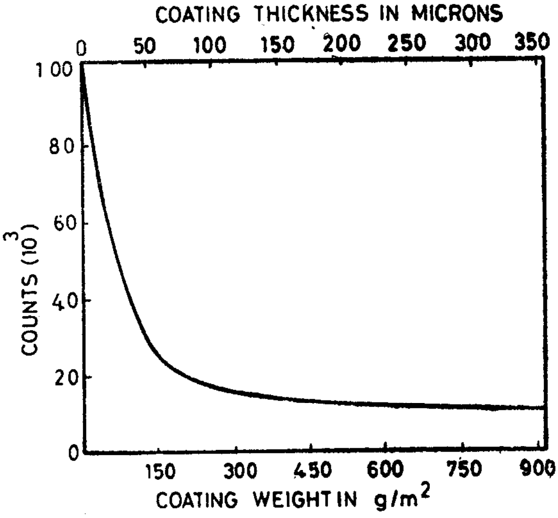


FIG. 1 CALIBRATION CURVE SHOWING THE INTENSITY VARIATION WITH THICKNESS OF ZINC COATING USING AM-241 RADIO-ISOTOPE

in passing through the coating. Depending upon the atomic number of coating, when the coating thickness is increased to a certain value the characteristic radiation from the substrate disappears although a certain amount of scattered radiation will still be detected. The measurement of a coating thickness by X-ray absorption is not applicable if an intermediate coating layer of another metal is present because it will affect the absorption intensity. The typical relationship between coating thickness and intensity of a characteristic emission from the substrate material is shown in Fig. 2.



Aluminium

$N = 10 + 90\, e^{-3.51x}$

$\epsilon = 0{\cdot}326\ m^2/Z$

$\frac{1}{3} = 86{\cdot}88\ g/m^2$

$P = 2{\cdot}7\ g/cm^3$

FIG. 2 COUNT-RATE VERSUS COATING WEIGHT FOR ALUMINIUM COATING ON STEEL (IRON X-RAYS — X-RAY ABSORPTIOM METHOD)

5 FACTORS AFFECTING ACCURACY

5.1 Surface Cleanliness

Foreign materials such as dirt, grease or corrosion products will lead to inaccurate results. Protective coatings such as lacquer or heavy chromate conversion coatings over the coating to be measured will also affect the results. For all practical measurement purposes the errors arising due to above reasons can be considered negligible.

5.2 Specimen Curvature

Thickness measurement shall be made on flat surfaces if at all possible. Calibration standards having the same radius of curvature as that of test specimens can be used to eliminate curvature effects.

5.3 Temperature and Humidity

Coating or substrate temperature has no direct effect on gauge readings made using the XRF technique described in this method. Change in temperature and humidity occurring day to day have no effect on gauge reading.

5.4 Specimen and Measuring Apparatus Placement

Optimum reproducibility of results depends on proper positioning of the specimen relative to the measuring apparatus. The specimen should be positioned at the same point as the calibration standard used in calibrating the instrument.

5.5 Coating Thickness

The accuracy of the measurement decreases as the coating thickness approaches the limiting thickness as described in **4.1**. At heavy coatings the X-ray count rate changes very little with coating thickness and therefore the accuracy in that thickness region decreases.

The reason for this decrease in accuracy with increase in coating thickness is the exponential absorption of X-rays by coating material. Because of this effect the extrapolation beyond the thickness range established by the calibration standards can result in serious errors. As an example practical upper limit for thickness measurement of zinc coating is 460 g/m^2 per side.

5.6 Counting Statistics

The production of secondary X-ray Quanta takes place in a completely random manner. This means that during a fixed time interval the number of quanta emitted will always not be the same. This gives rise to the statistical error which is inherent in all radiation measurements. This error is independent of other sources of error such as those arising from mistake on the part of operator on the use of unreliable standards. To reduce the statistical error to an acceptable level it is necessary to use a counting interval long enough to accumulate a sufficient number of counts (at least 10 000).

5.7 Coating Homogeneity

Thickness determination by XRF method may be affected by the presence of foreign materials such as inclusions, co-deposited material, alloying, voids and porosity. The source of error will be minimized by using calibration standards obtained in the same conditions as those used in production of the coatings to be measured.

5.8 Substrate Composition

The effect of difference in substrate composition will be relatively minor on thickness measurements made by the X-ray emission method if the X-rays emitted by substrate are not hard enough to excite the radiation being measured. However, when thickness measurements are made by X-ray absorption method, the composition of the substrate of the calibration standard has to be the same as that of the test specimen.

5.9 Substrate Thickness

In using the emission method the substrate thickness shall be enough to prevent excitation of the coating from the reverse face on specimens coated on both sides. Failure to ensure this condition will introduce an error which will be large for thin coatings, becoming less and less as the coating thickness increases. However when thickness is to be determined by X-ray absorption technique the thickness of the substrate has to exceed a certain minimum or critical value which shall be established experimentally for a particular substrate-source combination. Substrate thickness in X-ray absorption or emission techniques has no effect when the thickness exceeds the critical value at which the primary radiation can no longer penetrate the substrate, be scattered from the matter behind the substrate and penetrate the substrate again which may cause additional fluorescence. For substrate not exceeding the critical thickness the effects may be minimized by backing the specimen with sufficient thickness of material of similar composition and density. The critical thickness of iron or copper substrate is approximately 0·1 mm.

5.10 Excitation Energy

The intensity of characteristic X-radiation either from the coating or from the substrate is strongly effected by variations in intensity or energy distribution of the primary radiation from the radiation sources.

When using a radioisotope as the source the energy and the enegy distribution of primary radiation are constant. The system must be corrected for intensity change as the radioactive material decays with its characteristic half-life. When using an X-ray tube as the source of primary radiation the output intensity can vary as a function of tube current, the average energy can vary as a function of anode voltage and the shape of energy distribution can vary as a function of conditions within the X-ray tube. The stability of X-ray tube should be checked after every 1 to 2 hours by the use of calibration standards. If instability of the radiation source is suspected, a series of ten or more measurements are made on the same specimen and standard deviation is calculated. This result should not be greater than the square root of average number of counts per measurement, N.

5.11 Detector

Errors can be introduced by an erratic operation of the detector system which is taken to include associated discriminator and scaling circuitary as well as the detector tube itself. If instability is suspected, a series of ten or more measurements are made on the specimen after which the standard deviation of the series should be calculated. The result should not be greater than the square root of the average number of counts per measurement, N. It should also be recognized that all radiation detection system has an upper limit with respect to reliable counting rate capability. Above this limit, a certain proportion of the total X-ray pulses will enter the detector during its recovery period or dead time and will not be counted. As a result, measure of true intensity will not be obtained. With large apertures in combination with high tube currents very high counting rates may be obtained. Manufacturer's instructions usually provide a procedure for determining the linear count rate capability of a particular detection system.

6 CALIBRATION

6.1 General

While taking readings for the purpose of establishing an instrument calibration, the same instrumental conditions including aperture size, voltage, measuring time shall be used as those which will be used on test specimen.

6.2 Standards

Reliable standards must be used in the calibration of the equipment, if accurate results are to be obtained. It should be understood that prolonged counting periods will not compensate for unreliable standards. Standards representing various thickness ranges of a number of coatings on different substrates are available with instrument manufacturers or can be prepared by electrogalvanizing or electroplating technique. If the chemical determination of the coating weight of the four satellite samples agreed to within about 2 percent, the control sample can be assumed to have a coating weight equal to the average of four samples and can be considered a good calibration standard.

6.3 A minimum of three standards covering the range of coatings to be measured shall be used for calibration. Because the calibration curve is non-linear, errors of interpolation may occur between calibration points. Extrapolation beyond the range of calibration points may also result in serious errors and should be avoided.

6.4 The instrument shall be calibrated with thickness standards having the same coating substrate as those being measured.

6.5 The coating of the calibration standards must have the same X-ray emission (or absorption) properties as the coating being measured. If the coating of the standards is under the same conditions as the coating to be measured the X-ray properties may be assumed to be the same for mass per unit area provided the specimen properties discussed in **5.7** are verified to be same for the standard and specimen.

6.6 If the thickness is to be determined by X-ray absorption technique, the substrate thickness of the standards shall have the same X-ray emission properties as that the specimen. This shall be verified by comparing the intensities of the selected X-ray emissions of both substrate materials in coated condition. In the X-ray absorption technique the substrate thickness of the specimen and the calibration standards should be the same unless the critical thickness is exceeded as defined in **5.5**.

6.7 If the curvature of the coating to be measured is such as to preclude calibration of a flat surface the curvature of the standard and that of the test specimen shall be same.

7 PROCEDURE

7.1 Instrument Operation and Calibration

The instrument shall be operated in accordance with the manufacturer's instructions paying attention to the factors listed in **5** and the instrument shall be calibrated in accordance with **6**.

7.2 Calibration Checks

Calibration check of the instrument, which is used non-continuously, shall be made each time before putting into service. This calibration check shall employ at least three standard samples covering the range of coatings to be measured (*see* **6.3**). Instruments used continuously such as in on-line operations shall be checked periodically to verify proper calibrations.

7.3 Precautions

7.3.1 *Substrate Thickness*

If the X-ray absorption technique is used, it is to be verified that the substrate thickness exceeds the critical thickness. If not, the calibration shall be made with a substrate having the same thickness and emission properties as the specimen.

7.3.2 *Measuring Area and Specimen Positioning*

The size of the measuring area will depend upon the size and shape of the specimen. In no case measuring area shall be larger than the coated area available on the specimen. The specimen shall be seated firmly and securely over the instrument opening. Manufacturer's recommendation shall be followed concerning the size of the standard sample. The distance between the measuring apparatus and the specimen shall be maintained same during calibration as well as testing.

7.3.3 *Curvature*

The proper measuring area shall be used to minimize the effects of curvature.

7.3.4 *Substrate Material*

The secondary emission from the substrate of the standard shall be the same as that produced by the substrate of the test specimen if the absorption technique is used and this shall be verified by actual tests. In case of a significant difference, new standards that agree with test specimen shall be used.

7.3.5 *Surface Cleanliness*

All foreign materials such as dirt, grease, lacquer, oxides and conversion coatings should be removed from the surface prior to the measurement without affecting the coating material. While making measurements specimen areas having visible defects such as flux, acid spots and dross shall be avoided.

7.4 Evaluation of Results

The conversion of intensity readings to the thickness units shall be done by the use of calibration curve determined for the particular combination of materials.

8 RECORD OF TEST RESULTS

8.1 The following data shall be included in the test report:

a) Technique adopted,

b) Primary radiation used,

c) Material of substrate and coating,

d) The calibration standards used,

e) Surface conditions of the coating,

f) Equipment used and settings,

g) Test results, and

h) Inspector's name and date of testing.

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